Keysight Technologies

Using Non-Contact AFM to Image Liquid Topographies

Application Note

Introduction

High resolution images of patterned liquid surfaces have been acquired without inducing either capillary wetting ("bridging") or strong perturbations of the liquid surface profile. Using a 5500 AFM system with Mac Mode® Controller from Keysight Technologies, Inc. in dynamic force mode an oscillating cantilever/tip ensemble is used to probe the liquid surface. In order to achieve high resolution stable images on these patterned liquid surfaces, it is crucial that the oscillating tip not contact the liquid surface. [1] The liquid topographies were imaged in non-contact, amplitude modulation AFM (NCAM-AFM), a dynamic imaging mode where the AFM cantilever is vibrated with relatively small amplitude A_{fr} (1 to 10 nm) at a fixed frequency v_0 which is about 100 Hz higher than the fundamental cantilever resonance frequency. When a sharp tip is brought sufficiently close to the surface, the attractive van der Waals force shifts the resonant frequency of the cantilever/tip ensemble to the left (decreasing). [2] Because the drive frequency of the cantilever is fixed, the amplitude of oscillation will decrease due to this shift. The software controls are set so that the approach terminates at a slightly reduced amplitude A_{sp} compared to A_{fr}. The AFM feedback mechanism is then activated in order to maintain this reduced amplitude A_{sp} by adjusting the height of the tip relative to the sample surface. The tip is then raster scanned over the surface with the feedback loop enabled while monitoring the height of the tip. This data is then used to create a topographical image of the surface.

The non-contact mode is maintained only for a small range of A_{sp}. Decreasing A_{sp} moves the tip closer to the surface, increasing the lateral and vertical resolution, but also increasing the risk of accidental contact between the tip and sample. This can cause the interaction of the tip and sample to shift from net attractive (non-contact) to net repulsive (intermittent contact).

Figure 1 shows the switch from net attractive to net repulsive between region B and region A. The most easily distinguished attribute to this switch is the change in the phase response of the system. During the net attractive portion of the plot, the phase signal slowly decreases as the amplitude decreases. At the point where the system switches from net attractive to net repulsive, the phase signal makes a large positive jump and continues increasing as the amplitude is decreased by moving the tip closer to the surface.

Amplitude and Phase vs. Distance

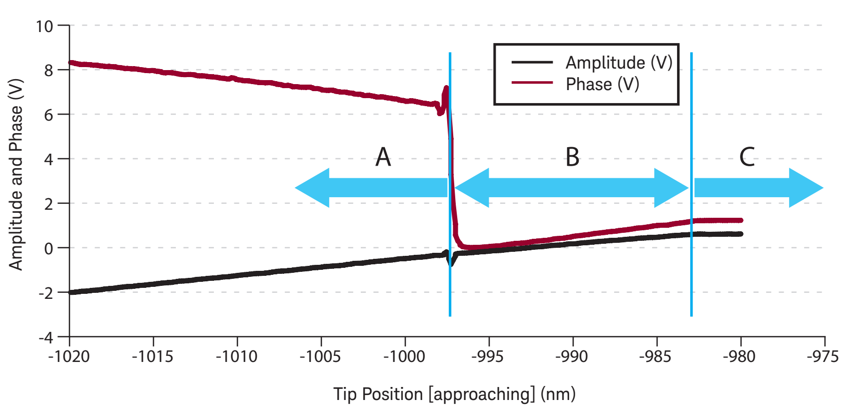

Figure 1. Amplitude and Phase vs. Distance (Z position of the tip) plot showing data for the tip approaching the sample. Region C has no interaction, region B is net attractive interaction, and region A is net repulsive interaction.

Figure 2 shows the switch back from net repulsive to net attractive that was acquired with the data from Figure 1. It is important to note that the transition on the retracting portion of the sweep does not occur at the same Z position as it does on the approaching portion of the curve. Because these transitions do not line up, the tip must be pulled away from the surface farther than the point where the transition from net attractive to net repulsive occurred in order to transition back to net attractive. The plots show data using a Nanosensors NCH cantilever with a spring constant of approximately 40 N/m and an initial amplitude of around 10 nm. The switch from net attractive to net repulsive occurred after the tip approached the surface 15 nm after the transition from no interaction. This can lead to unstable images with artifacts in phase and height data due to transitions between net attractive and net repulsive. The distance that the tip can move before the transition from net attractive to net repulsive can be increased by reducing the drive amplitude and thereby reducing the actual vibrational amplitude of the tip.

The data presented in Figure 3 shows amplitude and phase vs. distance data for the same cantilever that was used to acquire the data in Figures 1 and 2. The drive amplitude was decreased to 1/2 the value that was used previously and the input gain for the MAC Mode controller was increased by 2. This reduction in amplitude reduces the energy in the system and the tip has to be closer to the sample before the transition from net attractive to net repulsive occurs. This will improve image quality by reducing or preventing transitions from one regime to the other.

Amplitude and Phase vs. Distance

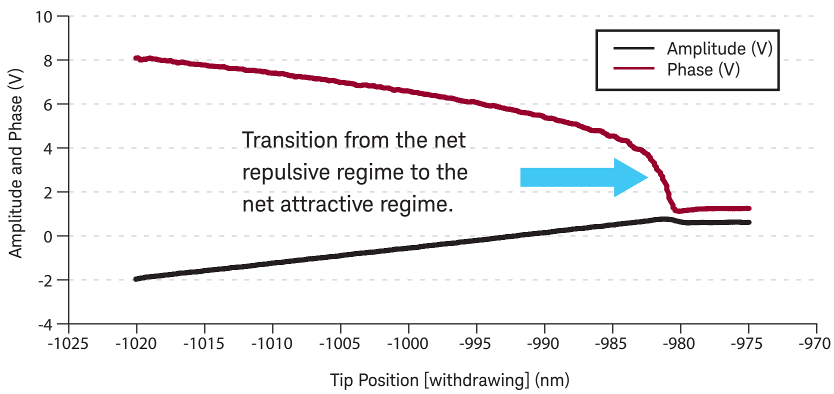

Figure 2. Amplitude and Phase vs. Distance (Z position of the tip) plot showing data for the tip withdrawing from the sample. The transition from net repulsive to net attractive is indicated.

Amplitude and Phase vs. Distance

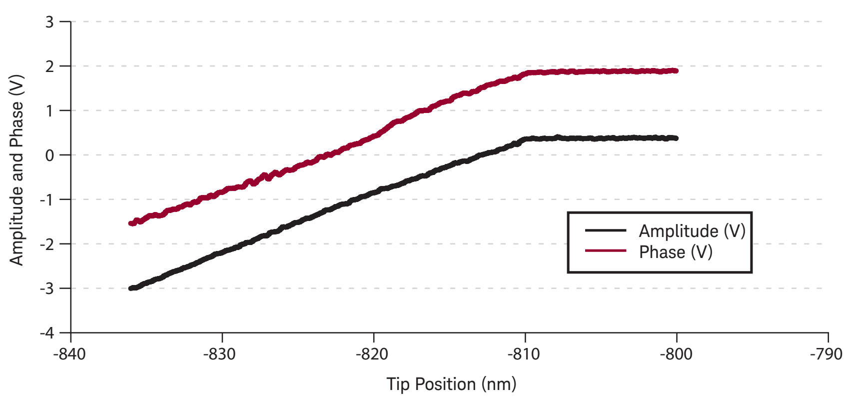

Figure 3. Amplitude and Phase vs. Distance (Z position of the tip) plot showing data for the tip approaching the sample. The drive amplitude for this plot was 1/2 what was used in Figure 1 and the gain setting for the AC Mode input was X2 instead of X1. The top curve is phase and the bottom curve is amplitude.

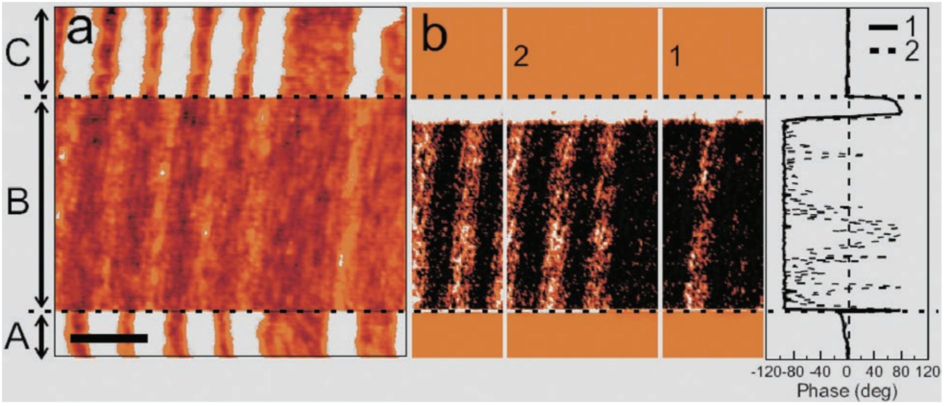

Figure 4. (a) Topography and (b) phase signal on “liquid nanostripes” change drastically as the setpoint amplitude A_{sp} is reduced (the lateral scale is 150 nm). Region A corresponds to NC mode where the topography of the liquid on the stripes can be discerned. In region B A_{sp} is reduced inducing a transition to IC mode and subsequent loss of liquid topography and a “phase jump”. In region C the original NC mode is restored. [3]

Guidelines for NCAM-AFM Imaging:

1. Choose a cantilever with a high spring constant value. This will help to maintain stable imaging conditions when using small drive amplitudes.
2. Choose drive amplitudes that keeps the oscillation amplitude at resonance small. Ideally, the actual amplitude will be quite a bit smaller than 10 nm.
3. Set the drive frequency to be 100 Hz higher than the resonant frequency of the cantilever. Zoom in on the Amplitude vs. Frequency plot to a range smaller than 5 kHz around the peak to make sure that the resolution is good enough to accurately determine the value of the cantilever resonance. This will ensure that the attractive regime interactions will reduce the amplitude of the cantilever oscillations as the resonant frequency of the system decreases.
4. Use an input gain of X2 or higher in the AC Mode Control dialog. This will effectively reduce the response of the system to changes in the setpoint.
5. When performing amplitude vs. distance spectroscopy, ensure that the limit function is utilized so that the tip does not crash into the surface. Typical values will be -1 or -2 for the V limit. This will protect the tip from damage and help to maintain the highest possible resolution.
6. Keep the sweep range as small as possible to minimize the motion of the tip relative to the surface. This also reduces the tip velocity for a given sweep duration which makes the interactions more stable.
7. While imaging, use a slower scan rate to allow the tip to move over the surface at a lower velocity. This will keep the tip sample interactions more stable and will result in less of a change in amplitude as features on the surface are encountered which will help to ensure that there are no transitions from net attractive to net repulsive tip sample interactions.

Things to avoid for NCAM-AFM Imaging:

1. *Soft cantilevers.* Soft cantilevers will be more susceptible to becoming “stuck” in the fluid layer on the surface.
2. *Large drive amplitudes*. Larger drive amplitudes will cause the tip to push into the fluid layer which can cause the tip to image the substrate under the fluid or cause the tip to become contaminated by the fluid. It can also cause the fluid to be “wicked” away from the surface.
3. *Setting the drive frequency less than the cantilever resonance frequency.* Setting the drive frequency, for example, 300 Hz less than the cantilevers resonance frequency will almost always cause the tip sample interaction to occur in the net repulsive regime so the tip will be making intermittent contact with the surface on each cycle. This will drive the tip through the fluid layer on the top of the surface.

Notes:

1. S. Herminghaws, A. Fery, D. Reims, Ultramicroscopy 69 (1997) 211.
2. Y. Martin, C. Williams, H. Wickramasinghe, J. Appl. Phys. 61 (1987) 4723.
3. A. Checco, Y. Cai, O. Gang, and B. Ocko, Ultramicroscopy, Proceedings of the Seventh International Conference on Scanning Probe Microscopy (July 2005).

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